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E.J. Anthony

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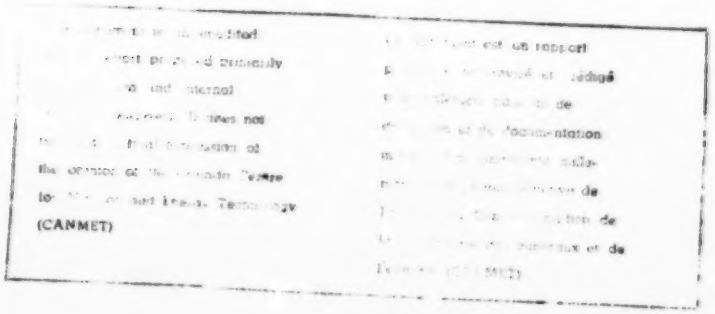

THE PRESENCE OF CALCIUM SULPHIDE IN SOLID WASTES FROM CIRCULATING FLUIDIZED BED COMBUSTORS

by

E.J. Anthony*

ABSTRACT

The presence of relatively high concentrations (≈6%) of CaS in solid residues from a sub-scale circulating fluidized bed combustor operated by the New Brunswick Research and Productivity Council (RPC) has raised concern that fluidized bed residues may be more hazardous than previously thougnt. In order to provide a better base of information, residues from a variety of bubbling bed and circulating bed combustors have been analyzed for CaS.

It was found that residues from bubbling bed combustors burning high sulphur fuels in the presence of limestone contain only minor concentrations of CaS (<0.2%). It is concluded that CaS can be generated in the hot cyclone and return leg of circulating bed systems, but that the high levels found in the RPC unit are probably atypical. For a number of reasons, residues from commercial-scale circulating bed combustors are likely to contain levels of CaS comparable to those found in bubbling bed combustors.

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*Research Scientist, Combustion and Carbonization Research Laboratory, Energy Research Laboratories, CANMET, Energy, Mines and Resources Canada, Ottawa, K1A 0G1.

PRÉSENCE DU SULFATE DE CALCIUM DANS LES RÉSIDUS SOLIDES PROVENANT DES INSTALLATIONS DE COMBUSTION À LIT FLUIDISÉ CIRCULANT

par

E.J. Anthony*

RÉSUMÉ

La présence de concentrations de sulfate de calcium relativement élevées (~6 %) dans les résidus solides provenant d'une installation de combustion à lit fluidisé dont les dimensions étaient inférieures à l'échelle, exploitée par le Conseil de recherche et de productivité du Nouveau-Brunswick (CRP), a remis en cause le problème voulant que les résidus provenant de lits fluidisés soient plus dangereux que prévu. Dans le but d'obtenir des renseignements plus précis, les résidus provenant d'un certain nombre d'installations à lit bouillonnant et à lit circulant ont fait l'objet d'une analyse visant à déterminer la présence du sulfate de calcium.

On a découvert que les résidus en provenance d'installations équipées de lits bouillonnants qui brûlaient des quantités considérables de combustibles sulfureux en présence de calcaire ne contenaient que des concentrations infimes de sulfate de calcium ($<0,2$ %).

Il est possible de conclure que le sulfate de calcium peut être produit dans la branche de retour et le circuit du cyclone à chaud des installations à lit circulant mais que les concentrations élevées de sulfate de calcium retrouvées dans l'unité CRP sont probablement atypiques. Pour toutes ces raisons, les résidus provenant d'installations de combustion commerciales à lit circulant devraient vraisemblablement contenir des niveaux de sulfate de calcium comparables à ceux retrouvés dans les installations de combustion à lit bouillonnant.

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*Chercheur scientifique, Laboratoire de recherche sur la combustion et la carbonisation, Laboratoires de recherche sur l'énergie, CANMET, Énergie, Mines et Ressources Canada, Ottawa.

INTRODUCTION

In March 1985 Dearborn Environmental Consulting Services was awarded a contract by the Canadian Electrical Association (CEA) to carry out characterization studies of solid wastes from circulating fluidized bed (CFB) combustion. The project's prime objective was to combust a wide range of Canadian solid fuels with limestone in a pilot-scale atmospheric CFB unit in order to generate representative solid waste samples and then to characterize these wastes to produce a database on their properties. Supplementary objectives were to identify potential areas of concern regarding the management of such wastes and to recommend areas requiring further research.

The Dearborn proposal called for the wastes to be generated in the pilot-scale CFB facility owned by GA Technologies Inc. in San Diego, California. However, by January 1986, it had become apparent that GA Technologies would not be able to complete the pilot scale work within the time frame set by the CEA project. Consequently, Dearborn subcontracted the pilot-scale work to New Brunswick Research and Productivity Council (RPC) which had recently commissioned a new 130 mm (5 in.) diameter CFB combustor.

The actual testwork was carried out by RPC in March 1986 and involved 10 successful combustion trials (subsequently a longer duration trial was carried out) with combinations of four coals and three limestones. The test matrix is given in Table 1 and the actual operating conditions are given in Table 2.

Upon subsequent analysis of the test results it became clear that the calcium utilization was at least comparable to typical bubbling bed performance. Furthermore, significant quantities of calcium sulphide (1-6%) were found in the solid residues. It is to the issue of calcium sulphide formation that this report is addressed, since its presence in significant quantities in the solid waste would make utilization problematic and increase disposal costs.

EXPERIMENTAL RESULTS FROM RPC

Table 3 gives the chemical composition of the various samples taken from the RPC rig during the test series. The designation B refers to bed material sampled from a standpipe in the J valve section of the combustor

(see Fig. 1) while BH refers to materials taken from the baghouse. The $CaSO_4$, $CaCO_3$ and C were determined by wet chemistry, the CaO by means of both X-ray fluorescence and neutron activation and the CaS was calculated by difference from the analysis of total sulphur and sulphate sulphur, the presence of CaS having been confirmed by X-ray diffraction (1).

The most striking fact about the results in Table 3 is the very high concentrations of CaS in the ash residues (up to 6%). Secondly it is apparent that except for runs 7 and 10 most of the CaS is found in the bed material, while CaS percentages in the baghouse ash are typically 1% or less. This suggests that the origins of the CaS are likely to be associated with the return leg of the RPC combustor rather than reactions occurring in the bed itself or the baghouse. Samples S-B-11 and S-BH-11 were obtained from a separate set of combustion tests carried out with Minto coal, Albert County oil shale and limestone in a pilot-scale CFB in Frankfurt, Germany owned by Lurgi GmbH. The analytical results for these samples support the thesis later developed in this report that CaS if formed, is substantially eliminated in large CFB combustors. However, in order to understand the processes occurring, it is worth examining the literature on CaS formation and its presence in FBC systems.

FORMATION OF CaS

The presence of CaS in wastes from solids in AFBC systems has been previously identified (2,3). However, the concentrations are so low that Sun et al. (3) states that it is not a problem for once-through FBC systems as opposed to systems with a sorbent regeneration feature.

Unfortunately, the actual amounts are not quantified. Nor have workers analyzing residues from Canadian AFBC systems quantified the CaS content. They have noted that its presence can be occasionally detected by smell as H_2S when acid digesting solid residue samples from AFBC systems (4,5). Since H_2S can be sensed by the human nose at the ppm level (6) this has not been judged as significant and no actual measurements for CaS have been made. Regarding CaS in CFB wastes, up to the time of writing there have been few or no sources of such wastes in Canada and no measurements were made prior to the CEA study (1).

In order to understand how CaS might be produced in FBC systems it is necessary to consider the chemistry involved. Older textbooks on inorganic chemistry describe the Leblanc process for CaS formations which involves heating calcium sulphate with powdered coke at 900°C and follows the stoichiometry (7):

$$CaSO_4 + 4C \rightarrow CaS + 4CO \qquad \text{Eq 1}$$

CaS can be produced at temperatures between 700°C-1000°C. However, at lower temperatures (700°C-800°C) it is produced in a reaction described by the stoichiometry:

$$CaSO_4 + 2C \rightarrow CaS + 2CO_2 \qquad \text{Eq 2}$$

while the first reaction predominates at higher temperatures (800°C-1000°C). It can also be produced directly by CO at temperatures as low as 680°C (8,9). However, at temperatures above 900°C the competing reaction:

$$CaS + 3CaSO_4 \rightarrow 4CaO + 4SO_2 \qquad \text{Eq 3}$$

becomes increasingly important and has subsequently been investigated as a method of regenerating sorbent in FBC systems (10).

CaS can also be oxidized back to CaO both by steam, if present in the temperature range 750°C-1600°C, and by oxygen to $CaSO_4$ or CaO according to the stoichiometry:

$$CaS + 2O_2 \rightarrow CaSO_4 \qquad \text{Eq 4}$$

$$CaS + 3/2\ O_2 \rightarrow CaO + SO_2 \qquad \text{Eq 5}$$

reaction (4) occurs at teperatures above 400°C, and reaction (5) dominates at higher temperatures (>950°C) (11).

In addition to all of the above reactions, calcium sulphate may also be directly reduced to CaO by C in the reaction:

$$CaSO_4 + 1/2\ C \rightarrow CaO + SO_2 + 1/2\ CO_2 \qquad \text{Eq 6}$$

This reaction and reaction (2) have been studied in detail by Turkdogan and Vinters (12) who have shown that with a C:S molar ratio of 2:1 the conversion of $CaSO_4$ to CaS can be almost quantitative with a loss of less than 1% as SO_2. Almost complete conversion to CaO occurs at C:S molar ratio of 1:2. They found that the reactivity of the carbon had a marked effect on reactions (2) and (6), thus graphite tended to favor the formation of CaO while more reactive species tended to favour CaS formation. Interestingly enough, their work suggested that the volatiles content of the coals did not affect the process, only the reactivity of the char.

FORMATION OF CaS IN BUBBLING BED VERSUS CIRCULATING BEDS

Despite the complexities of the reactions listed above, it may be easily explained why CaS might be formed in CFB but not to any significant degree in bubbling beds. There are basically four sources of solid residue from a bubbling bed combustor (see Fig. 2):

- the bed or combustor;
- the cyclone;
- the heat exchanger used to protect the baghouse;
- the baghouse.

In a bubbling bed system the bed will be in the correct temperature range for the reactions which produce CaS (700°C-1000°C). However, the environment is highly oxidizing and it is unlikely that significant quantities of CaS will be produced.

The cyclone used for fly ash recycle may provide a reducing environment, but bubbling bed systems do not use hot cyclones, therefore the temperature range will likely be between 300°C and 500°C, below that at which CaS is likely to form.

The heat exchanger used to protect the baghouse, dependent on the rig's design, may precipitate hot solids. However, since baghouses are designed to operate with solids at less than 200°C, the temperature regime in the heat exchanger is again likely to be below the 700°C ceiling for the reactions which produce CaS. In addition, the environment is quite likely to be oxidizing.

Baghouse solids will be at temperatures less than 200°C. At these temperatures no CaS can be expected to form.

CFB's have four potential sources of solid residues (see Fig. 3):

- the bed/combustor;
- the hot cyclone or return leg;
- the heat exchanger used to protect the baghouse;
- the baghouse.

In the case of the CFB, there may be no well-defined bed. However, in some designs, the combustor will have an expanded bed section at the bottom or only part of the combustion air will be introduced into this section allowing a bubbling or turbulent bed to exist.

It is unlikely that significant quantities of CaS will be produced in this section, although the temperature range is suitable (700°C-1000°C), because the bed solids are likely to be in an overall oxidizing environment.

The return leg (J or L valve) and the hot cyclone will be in the right temperature range (700°C-1000°C) and are also likely to provide a reducing environment in which $CaSO_4$ and coal char can be brought into intimate contact. It is expected that this region is responsible for CaS in CFB systems and this view is supported by the higher concentrations of CaS seen in the RPC "bed" samples which are in fact drawn from a standpipe in the return leg. In industrial systems, solids may well be withdrawn from this region either to remove oversized particles or to help maintain bed inventory, but the bulk of CFB solid residues normally come from the heat exchanger and the baghouse.

With regard to the heat exchanger and the baghouse sections both the temperature (<700°C) and the chemical environment (oxidizing) are unsuitable for the production of CaS. Thus any CaS found in samples from these sections will probably have originated from the hot cyclone return leg of the CFB. Concentrations should also be expected to be significantly lower by virtue of the particles having had to pass through the oxidizing regime in the combustor.

In order to test these arguments it was decided to obtain solids from bubbling bed and CFB systems in Canada or from systems used to test Canadian fuels. A secondary aim was to provide a cross check on the chemical techniques used to determine the CaS in the RPC samples for Dearborn.

SOURCES OF FBC SOLID RESIDUES

Samples were obtained from three bubbling bed systems:

- Queen's pilot-scale AFBC (13) (burning Devco Prince coal and using Havelock limestone).
- Point Tupper 1 m^2 pilot-scale AFBC rig (14) (burning Lingan coal and using Irish Cove limestone - samples taken from run 10, the last 1000-h segment of the corrosion trials).
- The Canadian Forces Base, Summerside industrial fluidized bed boilers in Prince Edward Island (15) (burning Devco Prince and using Havelock limestone).

In addition, samples were obtained from two circulating bed facilities:

- The 100 mm diameter bench-scale reactor at CANMET's Mineral Sciences Laboratories (MSL) (16) (burning Syncrude coke and using Athabasca limestone).
- The Hans Ahlstrom 600 mm diameter test facility in Finland (17) (burning Syncrude coke and using Athabasca limestone).

Two of the original samples from the CEA study were taken and analyzed as a cross check on their chemical techniques.

EXPERIMENTAL METHOD

The analysis was performed using a modification of the method of Kusenko et al. (18,19) and the semi-micro apparatus developed by Knott and Belcher (20) together with the Fisher sulphur analyzer. The method involved:

- evolution of H_2S from the sample by refluxing in glacial acetic acid;
- displacement of the H_2S by an inert gas stream;
- moisture absorption by passage of gas stream through a bubble tube filled with magnesium perchlorate;

- oxidation of H_2S to SO_2 by addition of oxygen to the gas stream and passage through a bubble tube filled with Schutze reagent;
- determination of SO_2 by the Fisher sulphur analyzer, Model 475.

The actual procedure was as follows:

- weigh 20 to 2000 mg of sample into a 3-neck, 100-mL boiling flask fitted with a dropping funnel, reflux condenser and inlet tube for the feeding of inert gas;
- attach to apparatus;
- start analysis cycle on sulphur analyzer;
- add 30-40 mL glacial acetic acid to reaction flask through dropping funnel;
- start flow of inert gas (~1 L/min);
- bring to a boil;
- start another analysis as quickly as possible after the end of the analysis cycle (3-4 min).

EXPERIMENTAL RESULTS

Table 4 gives the analytical results obtained for two of the RPC samples together with Dearborn's measurements. These results are in extremely good agreement, especially when it is realized that a technical grade of CaS, of uncertain purity, was used for standardization.

Table 5 shows the results for samples from the bubbling bed combustors at Queen's, Point Tupper and Summerside. These show that CaS is present in concentrations less than 0.2% and may be regarded as negligible.

Table 6 gives the results for the two circulating fluidized bed units burning Syncrude coke and Athabasca limestone. The first unit, that at MSL, has an expanded bed section and solids may be withdrawn from the bed, the heat exchanger and the baghouse.

As can be seen, the levels of CaS are comparable to those obtained in the baghouse material from RPC but not the very high levels seen in the "bed" solids obtained from the return leg of RPC's rig. One possible explanation for the difference between the two systems is that although the diameters and bed heights are similar (100 mm cf with 130 mm diameter and 3.0 m cf with 5.8 m high for the MSL and RPC facility respectively) the MSL reactor

does not have the same large solids inventory in the return leg. The return leg of the RPC combustor is charged with about 250 kg of sand at the beginning of each run and the recycle rate is about 35 kg/min permitting solids to remain in the return leg for 7 min or more. Thus bed solids from the MSL rig are unlikely to remain in contact with one another for the same extended period in a suitable environment for CaS formation as in the RPC combustor.

The results from the 8.5 m high, 600 mm diameter Ahlstrom unit were chosen from one of the longer runs which was deemed to be most like commercial operation. They indicate CaS levels which are almost as low as those seen in bubbling bed systems. The only exception is for solids from the recirculating loop, which corresponds to the return leg of the RPC unit from which bed solids were withdrawn for the CEA contract. Since solids would only occasionally be withdrawn from this region of a CFB, this suggests that the higher levels of CaS seen from the smaller pilot-scale rigs such as those at RPC or MSL are not likely to be representative of industrial and commercial systems. Instead, levels of less than 0.2% may be more typical.

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Table 1 - Test matrix for RPC combustion trials

Run	Coal	Limestone	Ca/S molar ratio
1	Devco Prince	Havelock	1.5
2	Devco Prince	Exshaw	2.0
3	Devco Prince	Elm Tree	2.5
4	Poplar River	Havelock	2.0
5	Poplar River	Elm Tree	1.5
6	Poplar River	Elm Tree	1.5
7	US Bituminous	Havelock	2.5
8	US Bituminous	Exshaw	1.5
9	US Bituminous	Elm Tree	2.0
10	Minto	Havelock	2.4
11	Minto[1]	Oil Shale/ Limestone	2.1
12*	Devco Prince	Elm Tree	2.5

*Long duration test (100 h).

[1] Test carried out on a Lurgi plot plant facility.

Table 2 - Operating conditions for CFB test work

Bed temperature		850 ± 25°C
Excess air		15-20%
Air distribution:	Primary	60%
	Secondary	40%
Gas velocity		5-8 m/s
Coal feed rate*		10-15 kg/h
Start up material		Silica sand mean size (1 mm)
Coal size consist		3.8 mm x 0
Limestone size consist		2 mm x 0
Test Duration**		Minimum of 12 h with stable air and fuel feed rates, temperatures and gas composition.

*Chosen to achieve desired excess air at the set operating condition.

**The test durations were recognized to be insufficient to achieve a bed turnover and were chosen on the basis of economic constraints.

Table 3 - Composition of the active chemical species in solid samples from RPC combustor

Sample	CaS	$CaSO_4$	$CaCO_3$	Free CaO	Carbon in baghouse
S-B-1	5.3	4.2	2.0	9.3	-
S-BH-1	0.4	8.8	1.8	14.6	30.8
S-B-2	1.8	0.8	7.1	1.0	-
S-BH-2	1.1	6.4	1.4	1.9	22.2
S-B-3	4.8	5.9	5.7	10.2	-
S-BH-3	1.3	16.7	1.6	15.2	15.0
S-B-4	3.0	2.4	2.5	15.1	-
S-BH-4	0.0	14.9	2.3	20.0	2.72
S-B-5	1.4	0.7	2.5	5.7	-
S-BH-5	0.0	12.1	5.0	11.0	5.4
S-B-6	0.9	1.3	0.2	8.1	-
S-BH-6	0.3	7.4	5.4	9.8	2.6
S-B-7	1.0	2.5	2.3	6.4	-
S-BH-7	1.3	8.2	4.8	9.4	22.1
S-B-8	1.4	2.6	8.9	0.7	-
S-BH-8	1.0	4.2	1.8	1.9	24.7
S-B-9	2.7	4.8	2.5	10.6	-
S-BH-9	1.0	8.9	2.3	5.6	19.2
S-B-10	2.5	0.7	2.3	5.5	-
S-BH-10	6.3	8.4	4.3	36.0	10.2
S-B-11**	0.0	17.8	1.8	2.3	-
S-BH-11**	0.0	12.5	1.1	*	-
S-B-12	3.8	6.7	2.7	14.6	-
S-BH-12	2.6	15.0	2.3	21.0	-

*Negligible.

**Analysis of residue from a Lurgi pilot plant facility burning Minto coal/oil shale and limestone.

Table 4 - Comparison of CaS analysis performed on RPC samples by Queen's and Dearborn

Reporter	Queen's	Dearborn
Sample No.	% CaS	% CaS
S-B-4	3.15	3.0
S-B-1	6.52	5.3

Table 5 - Analysis of bubbling bed residue samples for CaS

Sample origin	Run No.	Nature of sample	% CaS
Queen's pilot plant	850515	Bed material	0.0
Queen's pilot plant	850606	Bed material	0.03
CFB Summerside*	9:30-10:30 Nov. 24, 1983	Bed material	0.06
CFB Summerside*	9:30 Nov. 24, 1983	Bed material	0.18
Point Tupper facility	Run 10	Bed drain	0.02
Point Tupper facility	Run 10	Cyclone	0.02
Point Tupper facility	Run 10	Baghouse	0.0

*In the CFB Summerside design cyclone material is not withdrawn from the system.

Table 6 - Analysis of CFB residue for CaS

Sample origin	Sample designation	Source of sample	% CaS
MSL pilot-scale rig	A1099-86	Heat Exchanger	1.9
MSL pilot-scale rig	A1100-86	Baghouse	1.0
MSL pilot-scale rig	A1101-86	Bed material	1.1
Hans Ahlstrom reactor	A731-86**	Bed material	0.09
Hans Ahlstrom reactor	A732-86	Bed material	0.20
Hans Ahlstrom reactor	A733-86	Bed material	0.22
Hans Ahlstrom reactor	A734-86	Bed material	0.09
Hans Ahlstrom reactor	A735-86	Baghouse	*
Hans Ahlstrom reactor	A736-86	Recirculating Loop	1.7

*Undetectable.

**These samples are taken from run 16 of the Ahlstrom trials and involve baghouse recycle of 67%.

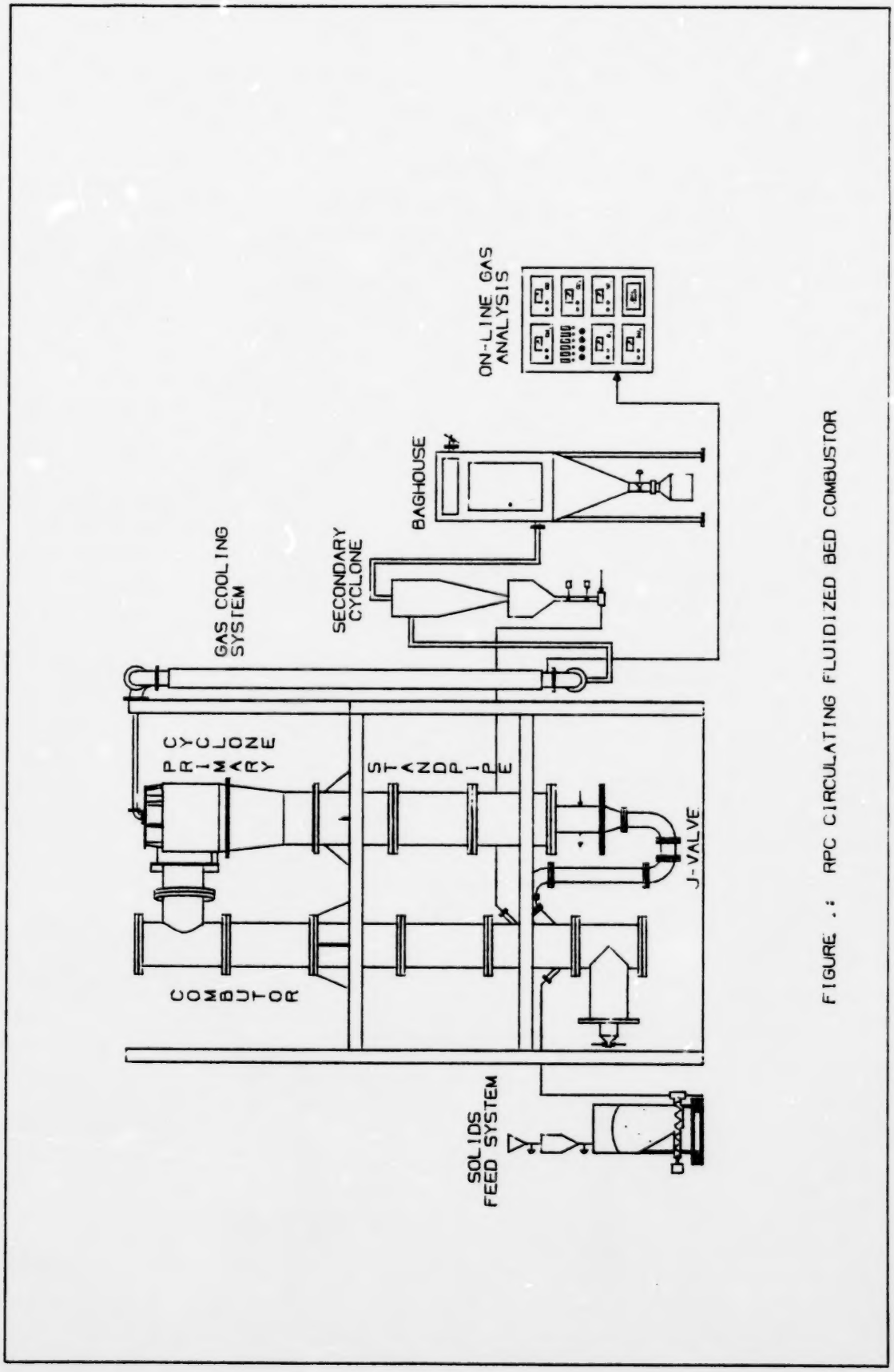
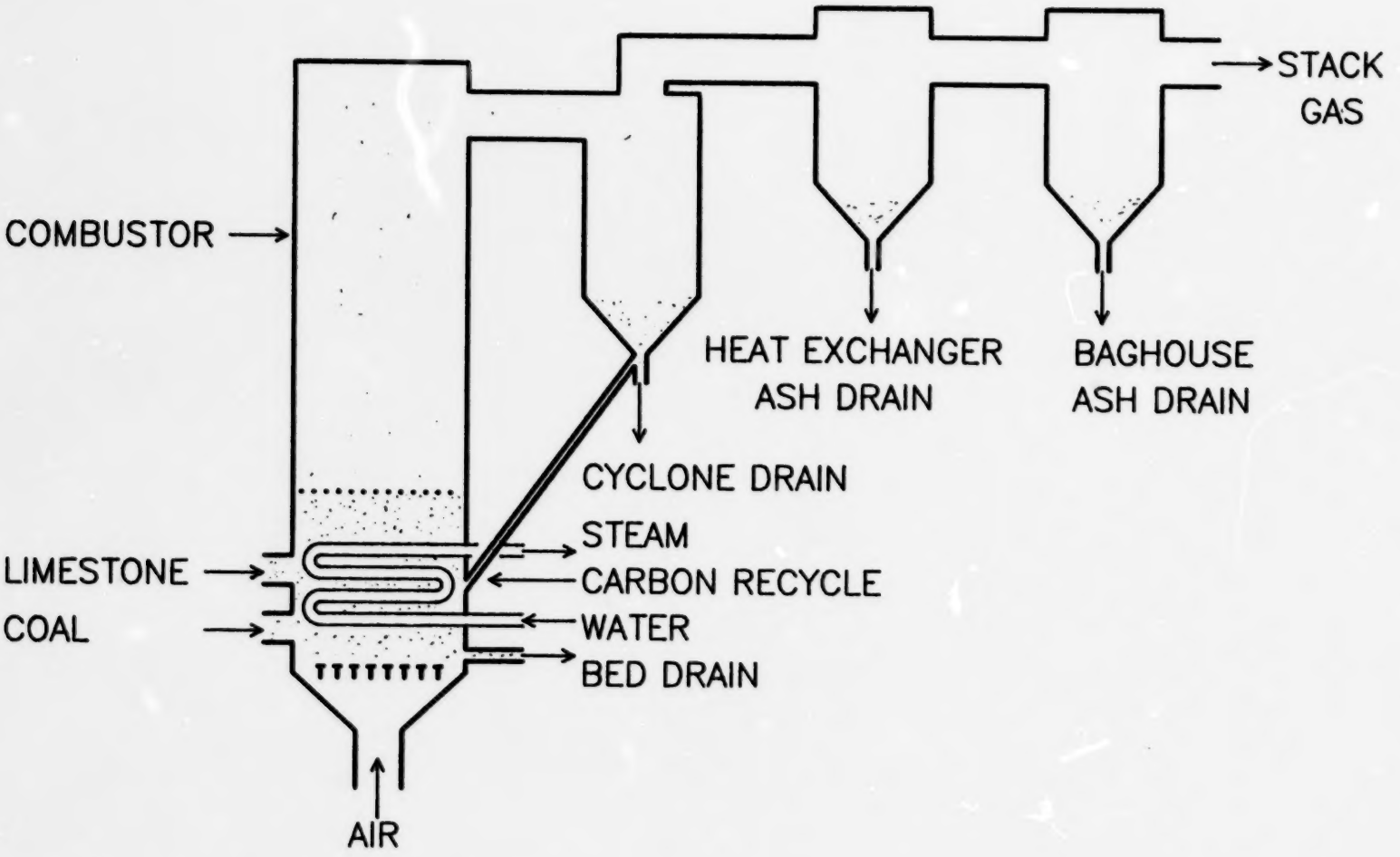
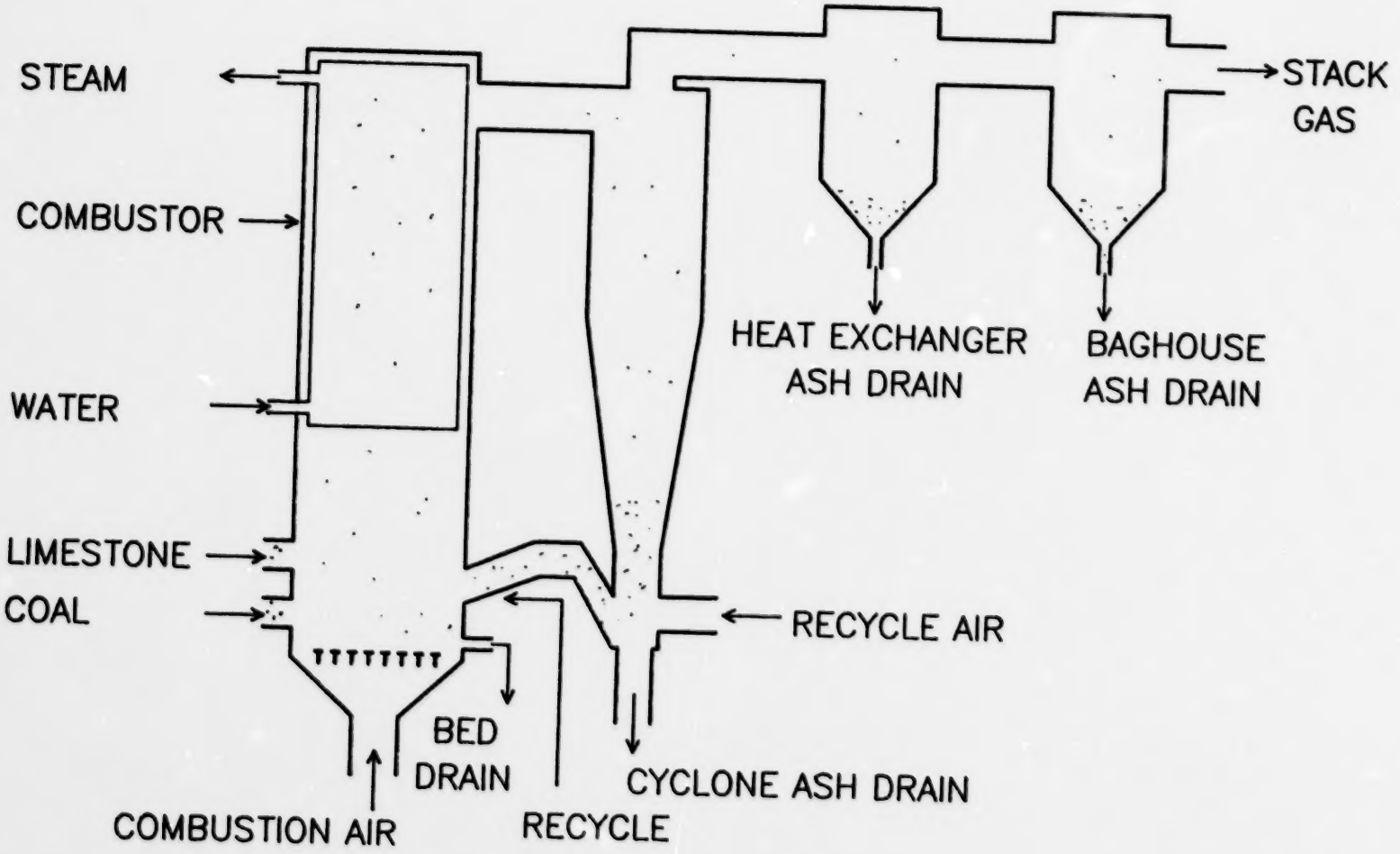


FIGURE .: RPC CIRCULATING FLUIDIZED BED COMBUSTOR



SCHEMATIC OF MATERIAL FLOW
IN AN AFBC SYSTEM

FIG 2



SCHEMATIC OF MATERIAL FLOW IN A CFB SYSTEM

FIG 3